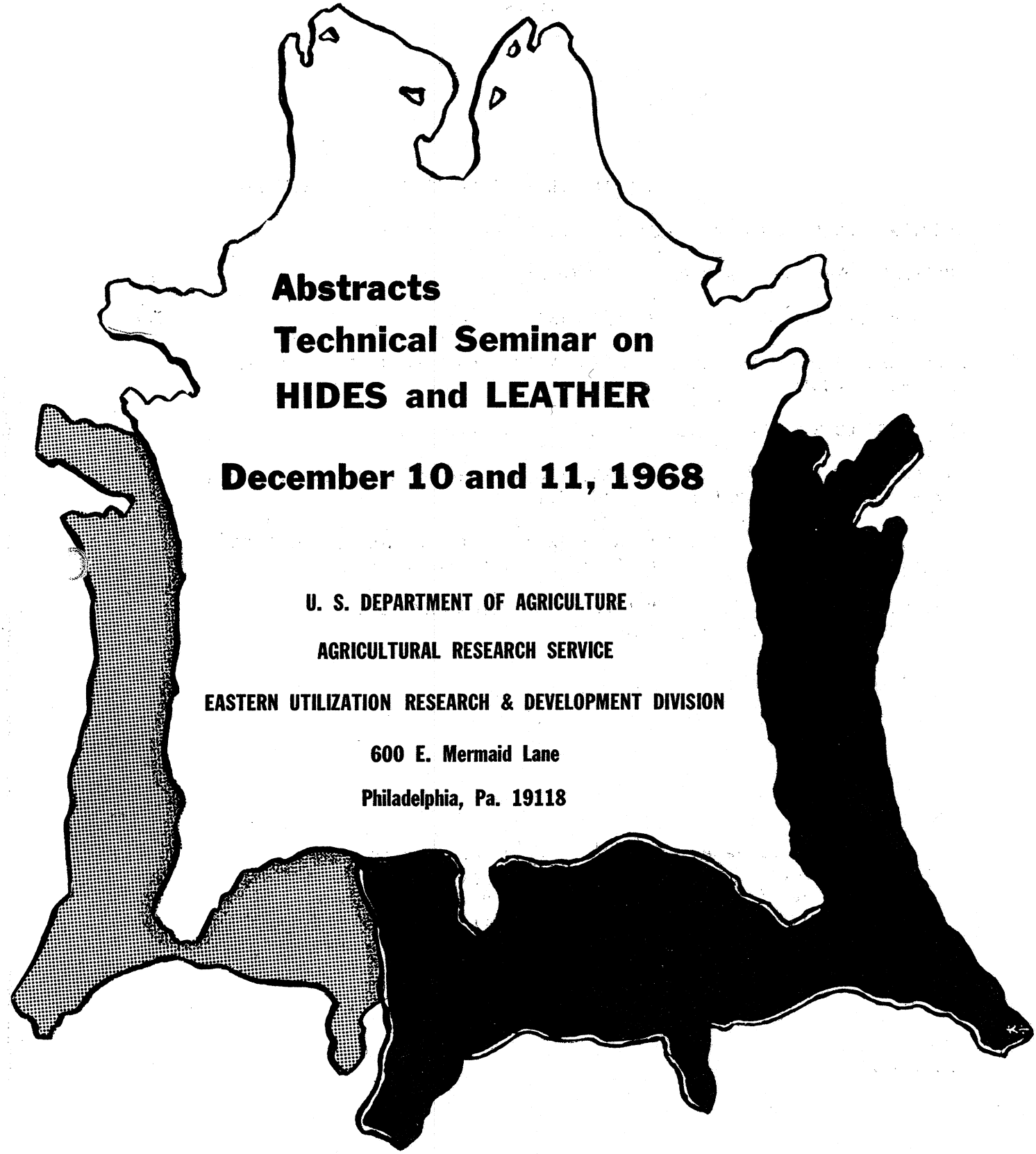

# Abstracts
# Technical Seminar on
# HIDES and LEATHER

## December 10 and 11, 1968

U. S. DEPARTMENT OF AGRICULTURE

AGRICULTURAL RESEARCH SERVICE

EASTERN UTILIZATION RESEARCH & DEVELOPMENT DIVISION

600 E. Mermaid Lane

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# TECHNICAL SEMINAR ON HIDES AND LEATHER

## WELCOME

by
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In the absence of Dr. Wells, Director of our Division, I should like to welcome the American Leather Chemists Association delegates to this technical seminar. We appreciate the advice and counsel of industrial visitors like yourselves as you review our research program for content, balance, and relevancy to current problems. We research people in Government need to be appraised periodically of industry's analysis of the present situation and its projection of present trends as they relate to our work. We are glad for the opportunity to present our research findings to you because success in our endeavors depends upon your eventual commercialization of our developments.

This is the second seminar we have held in cooperation with the ALCA. The first, held in the spring of 1965, was of great mutual benefit. We hope you find this one just as worthwhile, and that you will decide to continue the series.

## RESPONSE

by
Dominic Meo
American Leather Chemists Association
Liaison Committee on Research

The ALCA Liaison Committee on Research is glad to sponsor this second seminar. We look forward to sharing with you the problems we in industry are confronted with and are trying to solve. We appreciate that your Hides and Leather Laboratory, under Dr. Naghski's leadership, is the largest research group seeking solutions to our problems. We have a restricted representation of the industry here, since the seminar was set up on an invitational basis. Our hope is that these representatives will leave the conference thinking about what they can contribute to make your Laboratory's program even more valuable to their industry. Only a close working relationship between our own laboratories and your Hides and Leather Laboratory here at Wyndmoor will bring about the improvements in leather quality and greater efficiencies in operation that are needed to keep our industry healthy and in a strong competitive position.

We are sure that this second seminar will be as fruitful as the first one was, and we will be looking forward to others in the future.

# TECHNICAL PROBLEMS OF THE HIDES AND LEATHER INDUSTRY

by
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Armour Leather Company
Sheboygan, Wisconsin

Leather, whether it be for shoe bottoms, for shoe uppers, for distinctive garments, for upholstery, or for a myriad of other uses, possesses the characteristics desired by the consuming public. This has been so over the centuries, and today leather is in the most enviable position it has ever occupied. While we have been threatened over the years with substitute products, never has the genuine article been in such demand. There have to be reasons for this. I am firmly convinced that the consumer prefers leather over any other product developed to compete with it.

On the other hand, the worldwide consumer demand for leather and leather-like products is such that it does now--and will to an even greater extent in the future--exceed the possible worldwide supply of hides and skins. Therefore, it is essential to the well-being of all of us that better substitute products be developed and produced. If this does not occur--and I do not for a moment hold that as a possibility--there will be a worldwide shortage of these materials and consumer items made from them will be in greater and greater demand.

A posture of complacency in this industry could leave us in a vulnerable position. As these better substitute materials come on the market, it is of vital importance to improve the quality, the workability, and the desirability of leathers. These must be improved in esthetic areas, and here I use the terms eye appeal and feel appeal, that the consumer associates with leather quality and desirability. We must advance the feeling that leather is quality, quality is leather.

While the scientific nature of the product must be advanced, I seriously doubt the effectiveness of using technical descriptions or scientific discourse in the merchandising of consumer product utilizing leather. Rather the scientific knowledge should be couched in terms to create the impulse to buy. The average consumer is interested in the wear or life of a product, but does not really buy with this in mind. He or she buys the product because of its looks, its feel, or because it adds to his or her feeling of comfort, or perhaps more importantly, to his or her feeling of affluence.

Now what can the scientist in our industry do to effectively improve the nature of our product and to assure that leather will maintain its position of preeminence? First, we must improve our ability to maintain constant quality in the leather products or tannages that are developed. The key word here, I think, is reproducibility. I have been amazed and often discouraged to find that a product developed and successfully produced, promoted, and merchandised may occasionally lose some of its original quality. The tannage "goes off." Or to state it in still another way, some processing difficulty appears in the finished product. Too often as we analyze the problem we note the defect, but we are not able to determine what caused the failure to occur. Our general procedure at this point is to first blame the raw material, the season, the

humidity, or whatever else comes rapidly to mind. Finishing questions buffing, buffing questions fatliquoring, fatliquoring questions tanning, tanning questions the beam house, etc., etc.

When we have expended sufficient energies in these directions, we then begin to look at our process and our formulas. By and large, our technical people do an admirable job in this area. Loose practices are tightened, pH's scrutinized, time and temperature brought to more precise control. Shortly we are back "on the beam." Curiously, no one can now identify the original cause of the difficulty. The tanner points to corrections in the beam house, the finisher points to revised spray controls, and the guy from the lab notes that the hot, humid weather we had last week has, with the arrival of the cold front, moved on.

I have been somewhat facetious in my remarks on this subject. Certainly it is important for all of us to recognize the extremely complicated interrelationships that exist between the various processes that are employed as we alter the original hides and skins to make the desired end product. What can be done to improve our understanding of these complicated interrelationships? I recognize that this problem is not necessarily one for the industrial research laboratories, yet if all of us do not concern ourselves with the scientific investigation of problems, our progress will be pitifully slow.

Second, we must do all we can to enhance the natural beauty of leather. We have in our organization an individual who likes to keep this thought in front of all of us, and does so with the statement, "Paint is for houses; the natural, aniline dyes are for leather." Perhaps in the past we have put forth too much technical effort into an attempt to overcome or to hide some of the natural imperfections of leather. This effort has been directed to correcting and hiding such defects as healed scratches, fat wrinkles, and other blemishes.

Retaining and enhancing the natural beauty of leather is very important because our friends, the substitutes, can very easily come forth with imperfection-free surfaces. Some of the developers of the various synthetics are more aware of the merchandising pluses of the look of leather than are we. I frequently see synthetics designed and produced to contain ersatz healed scratches, veins, fat wrinkles, etc.

Third, after having developed the product and controlled its process to provide reproducibility, our technical efforts should be directed toward enabling it to retain its natural appearance and its desirable characteristics through the useful life of the end item itself.

Now frankly, I believe in planned obsolescence. Style and obsolescence go hand in hand. Therefore, it is not essential to produce a product with everlasting life. The desirable situation is somewhere in the middle. It is not sufficient to produce a piece of leather that will look attractive in the shoe or in the garment or in the furniture so that the consumer says "I want that," only to have that product fail early in its use either in appearance or in performance.

In upholstery leather great strides have been made both in appearance and in durability. Improvements have also been made in shoe and garment leather. There are some areas, however, where further advancement is needed. For example, we are all aware of characteristic failures occurring on frequent wetting and drying of leathers. We should also seek finishes that will not only enhance the natural beauty of shoe leather, but will give it two additional characteristics: somewhat greater scuff resistance (although an absolutely scuff-resistant leather for shoes may be unnecessary) and repairability (and I believe this to be the more important).

In the shoe business we can observe that new manufacturing techniques and new equipment and machines are beginning to play an ever more important role. A few years ago it was probably true that the shoe manufacturer more nearly adapted his manufacturing techniques to the use of leather, rather than the leather producer adapting his products to the techniques of shoe manufacturing. Partly as a result of style, but more importantly as the result of the need for efficiency and economy in manufacturing, the shoe industry is utilizing dielectric heat (with thermoplastic cement), Duo Process, or a similar seamless construction. This construction requires the use of lightweight materials to which the synthetics can very well adapt themselves. The leather industry is vulnerable.

The once-abundant supply of lightweight raw material for the production of lightweight or lined weight leather suitable for shoes is no longer available. Our livestock industry is operating on what might be known as a feedlot economy with a rapidly rising slaughter of bovine animals in the 900 to 1100 pound range. These produce a heavy hide. Concurrently we see a rapidly diminishing supply of calf, kip, and extremes, which for years have been a major source of the lightweight leathers used in the shoe industry. Not only are there fewer of the lighter bovine skins, but the supply of kidskins is also diminishing. Still, the need, the desire, and the demand for lightweight leathers will undoubtedly increase.

The challenge to the scientist, and also to the practical artist in the tanner, is to produce a <u>quality</u> piece of 2-1/2-ounce side leather, not out of unavailable raw material, but out of the hides flayed from feedlot steers and heifers. This means splitting down this raw material to an extent not now deemed feasible. The development of this quality of split-down leather is one of the most urgent tasks facing our industry today. Either we will learn to produce this quality piece of split-down leather, or we will ultimately toss this major segment of the shoe-leather market to the producers of synthetics. While this may be satisfactory for the shoe manufacturer and the producer of synthetics, it will not truly satisfy the consumer, or us as tanners.

There are other areas where vastly improved knowledge of shoe manufacturing requirements and techniques could be most useful to our industry. The ALCA-ASTM Joint Committee on Leather is working on some of these areas. I was interested to note that at a recent meeting the committee discussed and outlined some work to determine the correlation of the amount of oil in leather and its actual bond strength. This is an important area, and one in which our current level of knowledge is inadequate.

Some months ago, a new type of mulling operation was introduced to several shoe manufacturers in this country. This mulling procedure utilized alternately a fine water-mist spray followed by live steam, at correspondingly low and high temperatures. To the best of my knowledge, no shoe manufacturer came to a tanner to say that he planned to purchase and use a Brand X muller. Shortly after this piece of equipment was put into operation great hues and cries arose regarding the failure of the leather. It had blistered. After a great deal of expense and consternation, on the part of the shoe manufacturer and the tanner, the problem was recognized and ultimately accommodated, if not solved.

I am not in any sense critical of the shoe manufacturer. Rather I am critical of our industry for not being more acutely interested in and aware of the process changes he is making and the new equipment which is being designed and produced for him and which he is buying and using. It is incumbent upon our industry to become aware of these changes, and further, to design and develop leathers that accommodate themselves to these new manufacturing technologies.

Another area of changing components and technologies in the shoe factory that very definitely affects the leather is the use of thermoplastic materials for box toes, bindings, etc. We in the leather industry are not going to make, nor should we even contemplate, any effort to restrict the shoe manufacturers' inventive adaptations of these new technologies. We should, however, know a great deal more about the effect of thermoplastic materials on leathers so that we may develop products adaptable to these new technologies.

Again kudos to the ALCA-ASTM Joint Committee on Leather. Their formulation of a lastability committee is most commendable. As we develop leathers for the shoe industry, we are often well along the way before determining the lasting characteristics of that particular piece of leather. The result of this committee's work may provide us with useful data from which tests may be established to provide the lasting capabilities of leather as it is developed, rather than waiting for shoe-manufacturing trials or even more critical, actual shoe production.

What can you gentlemen do to help us in the area of color? I guess this is not a problem peculiar to the leather industry. Most industries utilizing color have frustrating problems. I have been told that this is true in the glass and ceramic industries, in the automobile industry, and even in the manufacture of refrigerators. We must, however, look at our problems.

Not many years ago when we developed, produced, and merchandised a color, subsequent lots of that color needed only to be in the general family of the originally adopted shade. This is no longer true. Our consumers, our retailers, and our manufacturers demand adherence to the original color specifications more closely than has ever been the case in the past. There are reasons for their demands. From a manufacturing standpoint, the stylist designing the shoe has chosen the color for his leather, then has had stripings, bindings, heels, knickknacks, etc. matched or coordinated with that leather color. All of these materials arrive from different sources. With the advent of computers, little lead time is provided. The materials reach

a central warehouse this afternoon and are dispatched to an outlying factory tomorrow morning. Cutting schedules have been prepared a week or two, or at least days, in advance. Machine time and labor is made available for the production of these shoes. When the components arrive at the factory, there must be no color deviation in any of them, and most particularly, they all must exactly match the original standard in the leather.

Is there not something that you gentlemen can do to standardize, not only at the tannery level but also at the shoe manufacturing level, a set of physical conditions that can be used to determine the color match? What can you gentlemen do to assist in the preparation of precise formulating methods for the reproducibility of a color, lot after lot after lot?

To these few suggestions, which frankly are oriented to the merchandising aspect of our business, should be added many well-known problems and objectives. Important problems await attention under raw stock, at the meat-packing plant and at the tanner. To cite a few, we have the cockle problem in sheepskin, grub eradication, brand damage, insect damage, effect of forced feeding on fiber structure, methods of preservation and curing of hides, new practices for marketing hides, and the all-important sewage-disposal problem.

There are many opportunities facing us--so many opportunities awaiting our endeavors and actions. What an interesting time to be associated with such an interesting industry!

## CHEMICAL MODIFICATION OF COLLAGEN

by
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This presentation consists of two parts, each about a chemical modification reaction of proteins developed at this Division. The first part concerns the reaction of a compound which has already been well established as a commercially important tanning agent--glutaraldehyde. The second part concerns a reaction which is, at present, of no commercial importance but still of considerable interest.

Much work has been done in this Division and in many others concerning the reaction of glutaraldehyde with proteins. Not only is it used as a tanning agent, but also as a fixative for microscopical studies of biological samples, as a precipitant in studies on the conformation and catalytic behavior of enzymes in the crystalline state, and as a disinfectant. All of these depend on its rapid reaction with proteins under extremely mild conditions. It is of some importance, therefore, to determine the nature of the reaction.

The first problem is to identify the reactants. For the protein, this is relatively easy. Amino acid analyses of several proteins before and after treatment with glutaraldehyde show that the amino acids which react are lysine, hydroxylysine, and tyrosine. The lysine is by far the most important.

There are complications with the other starting material--glutaraldehyde. The aqueous solutions of glutaraldehyde from different sources vary in the degree of purity of the glutaraldehyde. Perhaps the commonest type of impurity would be polymeric material formed by aldol condensations. This reaction would eventually lead to $\alpha,\beta$-unsaturated aldehydes, and there is evidence that these are present in some samples. These would also react with the protein; however, we have used samples of glutaraldehyde known to be free of these $\alpha,\beta$-unsaturated aldehydes and still obtained essentially the same reaction.

The main reaction has been shown to take place with $\epsilon$-amino groups of lysine (also hydroxylysine) residues in proteins as well as in model compounds, and gives the same three products on acid hydrolysis in all cases. The proteins that we have studied thus far are collagen and casein. The model compounds studied are $\epsilon$-N-carbobenzoxy-L-lysine and a poly-L-lysine having a molecular weight of about 80,000.

The glutaraldehyde-treated products after acid hydrolysis were separated by gel-filtration chromatography from each other and from other amino acids. The first product to elute is of relatively high molecular weight, is colored (brown), reacts with ninhydrin to give a purple color, and has little absorption in the near ultraviolet. This product is probably responsible for the color of glutaraldehyde-tanned hides. The second is nearly colorless, reacts with ninhydrin in the same fashion and has strong, sharp absorption at 265 m$\mu$.

This is the product which makes possible the quantitative test for glutaraldehyde in leather. The third is colorless, does not react with ninhydrin, and also has no absorption in the near ultraviolet. Work is in progress to determine the structures of these products.

The second modification reaction is the one we reported upon earlier, the reaction of proteins with malonic acid and formaldehyde. This reaction results in the addition of carboxyl groups to the protein by a Mannich-type reaction involving the basic groups of the protein, formaldehyde, and the active-hydrogen compound, malonic acid. This increase in carboxyl groups results in an increased capacity of the protein to bind metal ions in mineral tannages. The optimum increase obtainable of carboxyl groups is 35 percent as determined by a combination of three different methods.

Attempts to quantitate this reaction using amino acid analysis have been unsuccessful because only about 50 percent of the amino acids that have reacted can be accounted for in the form of new amino acids. Ten of these are easily detectable in hydrolyzates of collagen modified with formaldehyde and malonic acid, and two have been identified as $N_\epsilon$-($\beta$-carboxyethyl)-L-lysine and $N_\epsilon,N_\epsilon$-bis-($\beta$-carboxyethyl)-L-lysine. From model compound studies it has been determined which of the others are also derivatives of lysine, which are primary products, which are secondary products formed in the hydrolysis, and which are probably involved in crosslinking.

# COLORFAST DYES FOR LEATHER

by
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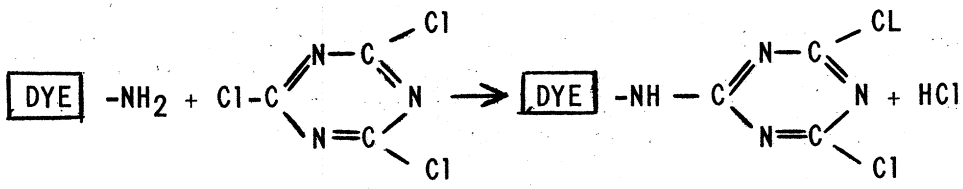
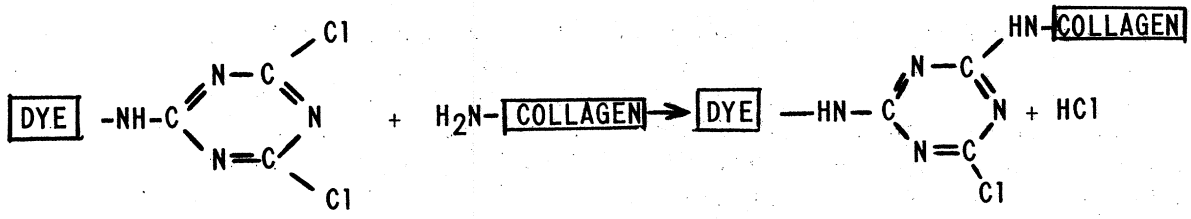
A natural corollary to the work done here on the development of a tannage that was resistant to washing was the application of wash-fast dyes to such leather in order to create truly washable leathers. During the many years that a variety of wash-fast dyes were made available for textiles, relatively few of them were found to be fast when applied to leather. By very special effort and selection, a number of standard leather dyes are fairly satisfactory if the leathers are not treated harshly. There are many complaints about staining, etc., but the consumer has come to realize that certain deficiencies and limits exist, and he has learned to accept them. However, with the advent of truly washable leathers, it is predicted that these deficiencies will no longer be acceptable.

During the past 12 years, various types of reactive dyes have been developed by dye manufacturers. Most were designed for use with textiles, especially cotton. Leather cannot withstand the conditions of temperature and pH required by most of these dyes. Until recently at least, and perhaps to this date, the Procion M dyes (ICI) comprise the only advertised series that will react under the mild conditions required by leather. The Procion M dyes are dichloro derivatives of s-triazine as shown below:




CYANURIC CHLORIDE PROCION M DYE




PROCION M DYE DYED LEATHER

The conditions required to break the dye-collagen bond would also probably break the polypeptide chain, and leathers dyed in such a manner would be bound irreversibly.

Glutaraldehyde-chrome combination tanned and conventional chrome-tanned skins, as well as glutaraldehyde-retanned leathers, were dyed with a series of Procion M dyes, according to the directions supplied by the manufacturer. The work on these skins has verified the results reported by British workers who showed these leather dyes to be fast to washing and drycleaning. For a series of eight dyes, using the ASTM-ALCA wash test, the degree of specimen fading was found to be extremely low. There is very little or no staining on the multifiber test cloth used in the wash test.

Over the years, the literature reports that leather colors fade during drycleaning. Garments must often be color-sprayed before they are returned to the customer. The British report the leathers dyed with Procion M are color-fast. Whereas the available British drycleaning test data is based on a standard laboratory test using perchloroethylene, the tests here were run on large pieces of leather or full skins in commercial drycleaning equipment.

Leather dyed with eight Procion M colors were drycleaned in each of three solvents.

Stoddard Solvent. The skins were agitated for 15 minutes in Stoddard solvent containing 4 percent detergent. This was followed by centrifuging and a 5-minute rinse in fresh solvent, followed by centrifuging again. The leather was tumble-dried for 30 minutes at approximately 145°F. (62.5°C.).

Perchloroethylene. The samples were agitated for 8 minutes in perchloroethylene containing 1 percent detergent followed by centrifuging. The leather was then tumble-dried for 20 minutes at approximately 150°F. (65.5°C.).

"Valclene" (1, 1, 2-trichloro, trifluoroethane). The skins were agitated for 3.5 minutes in Valclene plus 0.1 percent detergent. They were drained, rinsed in clear solvent with no detergent for 2 minutes, centrifuged for 2.5 minutes, and dried with room-temperature air for 6 minutes. This work was done in a special coin-operated machine developed for this drycleaning agent.

In no case was there any evidence of dye extraction into the solvent or staining on test cloth present during the process. The dyed leathers withstood the solvent with very little evidence of fading. After a second drycleaning cycle several test pieces seemed to be slightly paler on close examination. The change, however, would not ordinarily be considered significant in this field.

# FURTHER DEVELOPMENTS IN FREEZE-BRANDING

by
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Livestock improvement programs, disease control and eradication programs, and routine herd management are dependent on accurate identification of individual animals.

In recent years, the efforts to develop a better means of animal identification have been increased; however, to date we do not have an ideal system. Such a system would be one that:

1. Is permanent.
2. Is legible at a distance.
3. Does not cause pain or discomfort to the animal.
4. Does not damage the tissue or hide.
5. Is cheap and easy to apply.
6. Is not easily destroyed or lost.
7. Will conform to coding and data retrieval.

The most notable of the recent research efforts has been the work of Dr. Keith Farrell, Washington State University, who has developed a technique referred to as freeze-branding.

The basic goal of freeze-branding is to selectively destroy the melanocytes, or pigment-producing cells, responsible for the pigmentation of both hair and skin by the application of cryogenic materials. Destruction of these cells by freezing results in the growth of white hair in the frozen area. The complicating factor in this technique is to determine the right amount of exposure time necessary to destroy the melanocytes without damaging the skin and hair follicle.

In the fall of 1966, several preliminary freeze-branding studies were conducted at the Animal Husbandry Research Division, Beltsville, Maryland. Dairy and beef cattle ranging in ages from newborn to 18 months were freeze-branded on the rump and rib-cage area with 2- and 4-inch copper irons chilled in either a dry ice-alcohol bath or liquid nitrogen. The exposure times were varied from 5 to 30 seconds. After evaluating the results of these trials, we concluded that 30 seconds of exposure, using either dry ice plus alcohol or liquid nitrogen, resulted in both skin and follicular damage at these ages. There were differences in response between anatomical sites of the same animal as well as between animals. It was also apparent that age and breed differences are important in determining the optimum exposure time for freeze-branding. In addition, liquid nitrogen brands were inferior to dry-ice alcohol brands in legibility (white hair growth), and resulted in more severe skin and follicular damage across the range of ages and time exposures studied.

In an attempt to clarify these points as well as others, another study was

initiated in the spring of 1967. This project was designed to evaluate the results of branding with a 2-inch iron at 3, 6, and 12 months of age; chilled with two coolants (dry ice plus alcohol or liquid nitrogen); utilizing three time exposures (10, 20, and 30 seconds); on three anatomical sites (neck and shoulder area, rib-cage area, rump and thigh area); at three seasons of the year (spring, summer, and fall); in three geographic locations (east, south, west) with Holstein, Jersey, Guernsey, Hereford, and Angus cattle. The preliminary results of this study also indicate that the optimum time exposure depends not only on the coolant used, but the age at branding, the anatomical site selected, and the breed. Therefore, on the basis of these studies, we have made the following recommendations for freeze-branding dairy cattle when dry ice and alcohol is used to chill the irons:

1. Clip the hair as close as possible.
2. Remove loose hair, dirt, and dandruff.
3. Soak the area to be branded with alcohol.
4. Apply the iron with enough pressure to assure good contact.
5. Select the following time exposures by ages.

| Age | Exposure Time |
|---|---|
| a. Birth through 1 month | 10 seconds |
| b. 2-3 months | 15 seconds |
| c. 4-8 months | 20 seconds |
| d. 9-18 months | 25 seconds |
| e. Over 18 months | 30 seconds |

6. If liquid nitrogen is used, the above time exposures should be reduced by half.

7. An additional 10-15 seconds of exposure should be allowed on white animals. This will destroy the hair and result in a "bald" brand.

In summary, freeze-branding shows considerable promise of meeting many of the requirements desired of an ideal system of identification. For this reason, those of us in the cattle industry should make every effort to have it adopted as a permanent means of identification.

## A PROPOSAL FOR AN INTERNATIONAL SYSTEM OF ANIMAL IDENTIFICATION BY FREEZE-BRANDING

by
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A system of animal identification that utilizes freeze-branding has been developed. The proposed system consists of the 26 letters of the English alphabet, the numerals 0-9, and two crossbars. The 36 characters are uniquely designed to prevent alteration, yet are identifiable as the conventional symbols. The crossbars are arranged perpendicularly to form four quadrants which serve as a framework for the characters and assist in preventing alteration.

One of the 36 characters is placed in each quadrant in any one of eight positions (upright, inverted, side-right, etc.). The system is adaptable to computer processing in that the quadrants, symbols, and positions can be designated descriptively or numerically on standard computer cards. Over 27 billion unique brands are possible with this system.

The advantages of the proposed system are: (1) it provides a permanent, legible mark, (2) the application is painless, (3) alteration of the mark can be prevented or easily detected, (4) data on lost, stolen, or diseased livestock can be retrieved rapidly and efficiently, and (5) only a one-site mark is necessary.

## ALTERNATE USES FOR COLLAGEN

by
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About 2.5 billion pounds of cattlehides are produced annually. Most of these are made into leather, with minor quantities going into gelatin and glue. All these products are threatened by substitution.

Recent industrial research has resulted in the commercial manufacture of sausage casings and surgical items from hide collagen. The volume of hides consumed by these uses is expected to remain small. Work on reconstituted leather has virtually stopped, and we know of no efforts directed toward the large-volume use of hide collagen. Leather is still the main market. To us, there appears to be a large market for collagen in food. We have explored, for example, the feasibility of its use in meat products as a binder, extender, and coating, and have also considered it as a texturizer for vegetable proteins. Our efforts have been directed to developing collagen sources, working out processing techniques, and considering new possibilities for collagen-containing foods. We are also studying toxicology data, collecting information on the properties contributed to food by this hide protein.

We adopted limed cattlehide flesh splits as a low-cost source. Procedures were developed for deliming and for adjusting to a suitable pH to produce a product with a minimum of soluble solids. To make a meat product additive, the hide must be cut up to a particle size that will not interfere with the food's chewability. In a typical process, split sides adjusted to pH between 6 and 7 were cut in half lengthwise and fed to a strip cutter to produce 1/4-inch strips. These strips were re-cut to reduce their length. Alternatively, frozen pieces of split have been fed to a rotary knife granulator to produce pieces passing a screen of 3/8- to 1-inch aperture.

Either product was further subdivided by feeding to an Urschel Comitrol with a 0.060-inch head. The resulting product had about 30 percent solids and showed clean-cut hide particles with few loose fiber bundles and practically no reduction to fibrils. Mild treatment in a plate mill reduced about half of the particles to free, short fiber bundles.

Further mechanical dispersion has been made possible by reducing the pH of the 0.60-inch particles to 4.1 at 15 percent solids. Homogenization to an impalpable mass was accomplished in a Manton-Gaulin valve homogenizer at 1500 p.s.i. in each of two stages.

A third type of dispersion is made from ground or homogenized collagen (5 percent solids, pH 4.1) by warming in a high shear field in the Waring Blendor to a transition or denaturation temperature of about 55°C. where a sharp drop in viscosity occurs. This dispersion shows swollen fibrils in electron micrographs which lack the native 640 angstrom spacing, along with much amorphous material.

Collagen dispersions have been made less sensitive to heat by addition of fixatives. Dispersions that have been made cold form dry films that shrink after fixation. The shrink exhibited by films made from warm dispersions is less marked and is reduced further by addition of fixatives. A warm acid dispersion fixed as a dry film on a glass tube will not split open in boiling water. Effort is directed to the development of edible type fixatives.

When mixed with fresh ground beef, collagen granulated to 0.060 inch helps to retain weight, prevents loss of juices, and reducesshrinkage during frying or baking. Coarser granulation is detected in mastication. Finer dispersions become more difficult to mix. However, these may have a place as texturizers for vegetable proteins or as dispersants for unsaturated oils in simulated meat products.

Collagen is a non-allergenic protein lacking tryptophan and cystine and low in methionine. It has about half of the essential amino acid level of lean beef. Dry ground collagen was found to be completely digestible when fed to rats and was about 86 percent as effective as casein as a source of energy.

# AN ENGINEERING STUDY OF THE COMMINUTION AND CHARACTERIZATION OF LIMED FLESH CATTLEHIDE SPLITS

by
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A systematic study of the engineering aspects of comminuting rawhide for the efficient, large-scale production of collagen has been undertaken by the Engineering and Development Laboratory of the Eastern Utilization Research and Development Division.

The properties which contribute to the difficulty of cattlehide processing are its flaccidity, lack of dimensional stability, and susceptibility to heat denaturation. The product envisioned in this process is a fibrous slurry containing about 30 percent solids.

Limed flesh splits are delimed and cut lengthwise manually into four large strips. These are fed to a stripcutter which uses the scissor-like action of spinning blades and a stationary bed knife to produce smaller strips along with some relatively large pieces. This product is subjected to the chopping action of a rotary knife cutter, which also uses spinning blades and stationary bed knives, but with a one-inch screen. The material, now less than two inches in any dimension, is fed, along with powdered dry ice, to a high-speed grinder which produces a dough-like mixture of fibers and granules. Finally, the material is passed through a single revolving disc mill which provides the shear force necessary to separate the remaining granules into fibers.

Critical parameters of the material are, in part, water-binding ability, particle-size distribution, and protein denaturation. These parameters are being measured, respectively, by pH, by screening, and by viscosity and thermal analysis.

An industrial application of this process would, of course, include all necessary transport and control devices. Research is continuing on this and all other aspects of the project.

# ESTABLISHMENT OF KED INFESTATION IN SHEEP AS THE CAUSE OF THE COCKLE DEFECT IN SHEEPSKINS

by
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(EVERETT)

Cockle consists of a scattering of firm, brownish nodules or "pimples" in the grain layer of skins from wool-bearing sheep. Ordinarily it cannot be detected until the skin is removed from the animal and dewooled. The nodules vary considerably in size and arrangement, but they tend to form certain characteristic distribution patterns. When not severe, there are irregular patches in the neck and shoulders and light scattering elsewhere. The eruption spreads from front to rear and can cover the whole body, commonly forming rows in the mid-section perpendicular to the spine. Incidence of cockle rises sharply in the late winter and early spring, then falls abruptly by summer. Shearing hastens this regression.

The cockle defect seriously downgrades both grain and suede types of leather, being responsible for annual losses of about $4 million in this country. Although the problem has been worldwide for many generations, there is very little published research and no acceptable explanation of its cause.

In a preliminary survey, salted woolskins were obtained from a local wool-puller every month from December 1966 to May 1967. The skins were cut in half and only the left sides processed to the pickled stage. This permitted selection of matching right sides with appreciable cockle for further study in the fleece. A counting technique was developed using transmitted light on skins marked off in grid patterns. Data on the numbers and distribution of nodules confirmed established ideas about the incidence and severity of the defect. Total counts ranged up to 7,200 per skin, with fairly consistent symmetry between sides.

In the course of this study, repeated inspection of wool shorn from the selected right sides revealed higher numbers of keds (sheep ticks) in skins with heavy cockle than in skins with light cockle. Keds (Melophagus ovinus) are bloodsucking parasites and well-known pests of sheep, but were not thought to cause skin damage. Based on the experimental evidence and on consultations with Dr. I. H. Roberts, a new hypothesis was then developed implicating keds as the cause of cockle. The ideal facilities and willingness of Dr. Roberts led to planning a controlled test on insect-free sheep, half of which would be infested with keds in an attempt to reproduce a complete seasonal cycle of cockle in the test sheep.

(ROBERTS)

Mr. Everett has just revealed to you our conviction as to the causal relationship between cockle and the insect parasite of sheep, Melophagus ovinus, the common sheep tick, or ked. My purpose is to tell you a little about this parasite, and to summarize very briefly the experimental design on which our conclusions are based.

The common sheep ked is a bloodsucking, wingless fly. Singularly adapted for life on the skin and in the wool of its host, it is found in most parts of the world. The female does not lay eggs, but instead gives birth to larvae which are ready to pupate. The life cycle is completed in approximately 30 days. The female ked lives about 4 months, and produces approximately 15 larvae in that time. The parasites generally spend their entire lives on one host; when they spread it is usually by direct contact of the hosts.

Our experiment, begun in August 1967 at Albuquerque, New Mexico, involved 150 sheep, none of which had previously been exposed to keds. The animals were paired by age, weight, sex and, as nearly as possible, wool characteristics, and were separated into two equal groups. Five from each group were sacrificed in September, and their salted pelts shipped to the laboratory here in Wyndmoor; the skins were found to be essentially free of cockle, as anticipated. Beginning immediately thereafter, the 70 remaining sheep in the principal group were infested with 40 keds each, while the control group remained uninfested for the duration of the experiment.

As ked infestations increased from the original 40 keds to an average of nearly 300 during the following 8 months, equal numbers of principal and control sheep were sacrificed at 45-day intervals and their pelts were shipped here for study. Early in May, when the ked numbers were at their peak, there were 21 pairs of sheep remaining. At this point the sheep were subdivided into three groups, in two of which the keds were removed by shearing the animals or by dipping them in rotenone (a chemical known to destroy the insects) and in the third no treatment was used at all. Two subsequent slaughterings at 45-day intervals allowed us to determine, by examination of the skins, how much the normal disappearance of cockle would be hastened by removing the keds from infested sheep.

Data revealing the correlation between ked population and cockle spots will next be summarized for you by Mr. Everett.

(EVERETT)

Results of the test show conclusively that keds are indeed the causative agent of cockle. Skins from the sacrificed sheep displayed typical cockle with increasing severity as numbers of keds increased in their fleece. In its early stages the cockle was concentrated in the neck and shoulder regions; the keds appear to prefer this area. Later the entire body was affected as the keds spread out, and then the cockle regressed in summer as the keds succumbed to heat. Shearing and dipping appreciably hastened the regression by abrupt removal of keds. Some of the uninfested control skins exhibited "pseudo-

cockle"--blemishes closely resembling true cockle, but usually lighter in color and less extensive. The cause of these blemishes remains to be solved by further studies.

(ROBERTS)

In addition to the ked-cockle relationship, we assembled information concerning other injuries inflicted by keds, such as weight loss, carcass downgrading, carcass dress-out data (carcass weight vs. live weight), wool fiber damage, and changes in the blood-cell picture. These data will be presented at a future time.

I should like to conclude my presentation with a word concerning the control of keds. This parasite readily succumbs to a variety of pesticidal agents, applied as dips and dusts. A nationwide control program directed against the ked is not outside the realm of future consideration.

## RESEARCH AND CONTROL NEEDS IN RELATION TO HIDE DAMAGE BY ECTOPARASITES

by
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The role of ectoparasite research and responsibility for its funding and conduct, particularly in relation to hide damage, is not well defined or clearly directed. However, we recognize that most problems result from ectoparasite infestations. Among our 14 Divisional laboratories, at least four are concerned with studying internal and external parasites. Special emphasis on ectoparasite research is given at our laboratory in Albuquerque, New Mexico, directed by Dr. Irwin Roberts. Research studies at our other laboratories are primarily concerned with infectious diseases of all livestock species, including horses, and poisonous plant toxicology at Logan, Utah.

The research projects are identified under the current Planning-Programming-Budgeting System. This system gives the dollar amount of support for a given number of years, the location, the number of scientific man-years assigned, the probability of success, and a cost-benefit factor that reflects a saving justifying the research effort.

Once the research data have been developed and published, their application then becomes the responsibility of veterinarians, organized segments of industrial, county, State, Federal, and control groups, extension services, and others to use in formulating control measures. Such research is usually supported subsequent to economic losses from a given disease.

However, research data are not always applied in sufficient scope to make maximal use of the data. On the contrary, the approach may be fragmented and diluted nationally. In the long run, such a situation may be self-defeating as infectious disease agents do not necessarily respect county lines, state boundaries, or other political subdivisions. The motivation for research in food-producing animals has undoubtedly been deterred by lack of agreement concerning the significance of the lesion as a factor in the health of the animal. Hence, the incidence might be extremely high in the animals without cause for concern to the grower; yet, damage to hides, particularly in terms of quality, could be highly significant. This particular set of circumstances would not necessarily result in the initiation of research studies without emphasis from some segment of the industry. In the case of warbles, unfortunately, data currently available are not applied to develop maximal use of research data. No doubt there are degrees of indifference, misunderstanding, lack of coordination, or perhaps judgment about the seriousness of the various ectoparasitic problems that contribute to damage in hides. It is often felt that they have too low a priority to justify official regulatory control or to encourage the active promotion of funding for the needed research.

More precise methodology has resulted in the development of diagnostic techniques which have identified entities that can now be attacked on a broad front; namely, characterizing the cause of the disease, identifying the antibody, and developing data concerning transmission. These threats have literally swamped facilities. Scientists are not available in adequate numbers to attack all of these problems simultaneously on the broad front with a comprehensive team approach which, in most cases, is required to obtain valid data in the shortest possible time.

In the case of hide damage, we have a commodity problem inherited from another industry. It is an old problem, but apparently not one that the industry felt to be of sufficient importance to request and support nationwide control programs.

In the face of these many complexities, then, what are the courses of action left to the tanning industry in the solution of its problems? There has been no attempt here to imply that there is no further need for research. We recognize that, if additional research were conducted to permit easier and more effective methods of control, voluntary programs now in use would do more to reduce parasitism. Further research, however, would not necessarily cut down on those parasites (e.g., Demodex) which apparently have so little effect on the animal that the owner will not support research or make necessary expenditures for control. Therefore, hide damage would not be reduced.

If we had the interest and facilities of all of our research organizations and all of the groups concerned, we could quantitate the losses caused by these parasites, cite the research needs, and propose control programs. I am not attempting to oversimplify. Neither am I attempting to generalize, for we will not find the solution through generalization. Rather, I suggest that each problem be selected and evaluated independently, such as problems connected with Hypoderma bovis, Demodex, and other infestations. The problems should be characterized, perhaps by a committee from utilization research, the livestock industry, the tanning industry, the Animal Disease and Parasite and Entomology Divisions of Farm Research, the regulatory divisions, and the Extension Service. The Animal Disease and Parasite Research Division would be pleased to assist in delineation of the problems presented by ectoparasites. We intend to continue research on ectoparasites, at least at the current level. Hopefully, we may extend and expand such research when additional research data are needed to improve any segment of the livestock industry.

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|---|---|---|
| Aceto, N. C. | East. Util. Res. & Dev. Div., ARS | Wyndmoor, Pa. |
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